

A MICROSCALE COMBUSTION CALORIMETER FOR DETERMINING FLAMMABILITY PARAMETERS OF MATERIALS

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A microscale flow calorimetric method has been developed to measure flammability parameters of milligram samples under conditions which approximate flaming combustion. The present work seeks to improve upon a previously published thermogravimetric technique for determining flammability characteristics of polymeric materials [1, 2] by better reproducing the solid and gas phase processes associated with the flaming combustion of solid materials. The new microscale calorimeter accomplishes these objectives by rapid (>200 K/min) heating of milligram samples to a constant, calibrated heat flux in a thermogravimetric analyzer under an inert atmosphere followed by high temperature combustion of the pyrolyzate stream in excess oxygen. These processes correspond to the anaerobic thermolysis which occurs under non-oxidizing/reducing conditions to generate gaseous fuel and char in the pyrolysis zone of a burning solid and subsequent oxidative combustion in a diffusion flame.

Figure 1 is a schematic diagram of the microscale flow calorimeter. Mass loss is continuously monitored during the test in a thermogravimetric analyzer and volatile thermal decomposition products in the inert (nitrogen) purge gas stream are swept from the pyrolysis chamber through a heated transfer line and combined with oxygen in a heated manifold at the entrance to a coiled tubular combustion chamber maintained at several hundred degrees Centigrade. Complete oxidative combustion of the gaseous pyrolyzate occurs in the tubular reactor analogous to the combustion process in a well ventilated diffusion flame. The fuel/oxygen ratio and residence time in the combustor are simply controlled in the microscale flow calorimetric method through purge gas and oxygen flow rate adjustments so that incomplete combustion of the pyrolyzate stream is also possible. Complete and incomplete combustion products are scrubbed from the gas stream as it exits the tubular combustor. Heat release rate is calculated from oxygen consumption and mass flow rate measurements of the scrubbed gas stream as it exits the polarographic oxygen analyzer. Total heat released is obtained by numerical integration of the heat release rate and char yield is determined from the residual mass. Effective heat of combustion is calculated as the total heat divided by the mass loss, and the combustion efficiency from the effective heat of combustion divided by the chemical heat of complete combustion from an oxygen bomb calorimeter.

Figure 2 is a composite plot of the heat release rate data for some polymers tested in the microscale heat release rate device during 200 K/min heating to 50 kW/m^2 incident heat flux under nitrogen in the TGA furnace. The microscale heat release rate data are expressed as kilowatts per gram of original material in order to normalize the curves since the surface area of the sample varies between tests depending on the physical form of the material (e.g., powder, film, etc.). The polymers tested show a two order of magnitude range in peak heat release rate in the microscale device between the highest and lowest values.

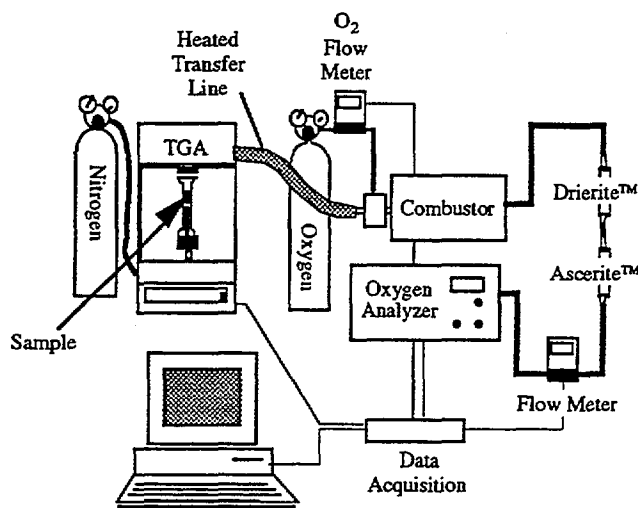


Figure 1. Schematic diagram of microscale calorimeter

Figure 3 compares the peak heat release rate data from the microscale heat release device to average heat release rate data from large (decagram) samples measured in a conventional cone calorimeter at 50 kW/m^2 incident heat flux for polymers where these data were available [3-7]. The cone calorimeter heat release rate values plotted in Figure 3 are steady-state or average values obtained by dividing the reported effective heat of combustion per unit area by the time of flaming combustion. Average heat release rate in the cone calorimeter is the bench scale fire parameter which should correlate with microscale calorimetric heat release rate if the steady-state heat release of thermally-thick specimens is simply a superposition

along the time axis of the isothermal heat release rate of the constant thickness, constant temperature pyrolysis zone as it moves through the sample during solid flaming combustion [8].

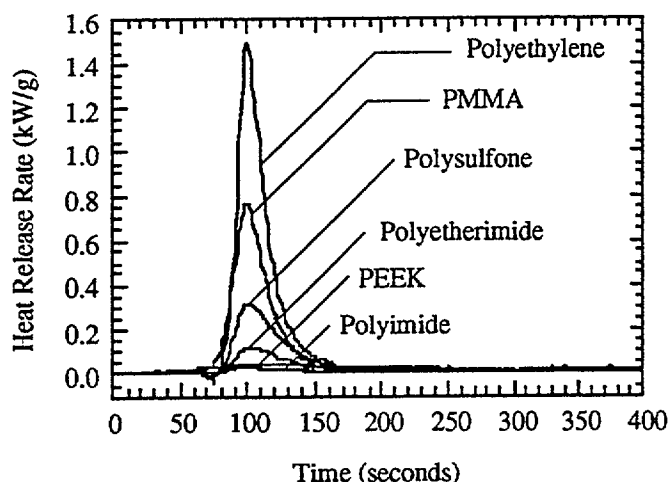


Figure 2. Heat release rate curves for various polymers at 50 kW/m² incident heat flux in the mini calorimeter.

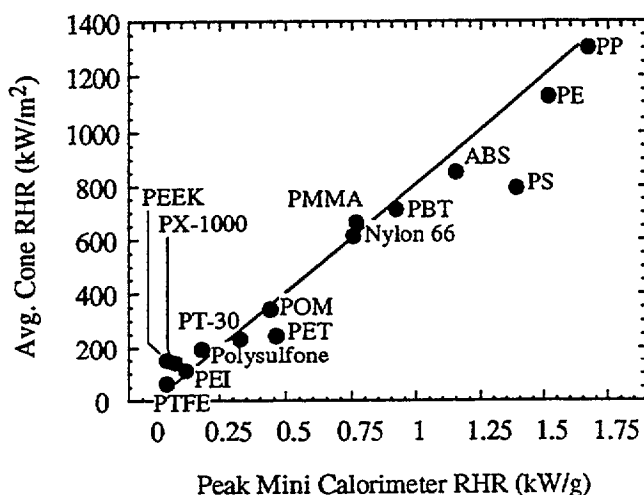


Figure 3. Correlation between polymer peak heat release rate in microscale device and cone calorimeter at 50 kW/m² incident heat flux.

Figure 3 indicates a good correlation between the peak heat release rates measured in the microscale device and the steady-state values measured in the bench scale cone calorimeter. As cone calorimeter heat release rate data appears to correlate well with full scale fire tests [9,10] microcalorimetric heat release rate should also be a good predictor of full scale fire performance.

Separating the thermochemical and thermophysical phenomena of flaming solid combustion in the microscale calorimetric method will more accurately reproduce the

local fire environment at the surface of a burning solid and should allow direct correlation with bench- and full-scale fire calorimetry tests. Decoupling of the intrinsic, time-dependent chemical processes of material combustion from transient effects associated with thermal diffusion in large samples. It is hoped that the new microscale combustion flow calorimeter will accelerate the development of new fire resistant materials by providing flammability parameters which are physically meaningful for use in fire hazard evaluation and will help relate material properties to full-scale fire performance.

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